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**SMALL ANGLE NEUTRON SCATTERING (SANS) CHARACTERIZATION OF
ELECTRICALLY CONDUCTING POLYANILINE NANOFIBER/POLYIMIDE
NANOCOMPOSITES**

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Abstract

Nanocomposites of polyaniline nanofibers and polyimide were fabricated and studied using small angle neutron scattering (SANS). The immiscible nature of the conformationally dissimilar polyaniline nanofiber and polyimide host is established by a series of experiments involving neutron scattering. Based on these techniques, we conclude that the crystal structure of the polyimides is not disrupted, and that there is no mixing between the two components on a molecular level. The morphology of the conducting salt component was analyzed by SANS data and was treated by two common models: Debye-Bueche (D-B) and inverse power law (IPL). Due to deviations in the linear curve fitting over a large scattering range, neither the D-B nor the IPL model could be used to characterize the size and shape of all PANI-0.5-CSA (polyaniline camphor sulfonic acid doped polymer)/polyimide blend systems. At 1 and 2% concentration, the D-B model suggested salt domains between 20 and 70 Å with fractal geometries implied by the IPL model. As salt concentrations increased to 5%, the structures were observed to change, but there is no simple structural model that provides a suitable basis for comparison.

Keywords: polyaniline, polyimide, small angle neutron scattering (SANS).

1. Introduction

Blending of polymers offers a means of engineering into one material certain combinations of desired properties exhibited individually by the component polymers. For instance, insulating polymers (such as polyimides) possessing many desirable technological properties may be rendered electrically conductive by mixing with conductive particles such as carbon black, metal powders, flakes or fibers, and metal-coated particles. The level of electrical conductivity in these heterogeneous materials depends primarily on the concentration and geometry of the conductive filler particles. The observed critical filler content which marks the onset of discontinuous increase in conductivity (i.e., the percolation threshold) in the majority of these composites falls between 15 and 25% (vol/vol). At such high filler concentrations, many of the desirable mechanical properties of the matrix polymer are either compromised or lost. However, when the conductive components are reduced in size from macro to nanosized, the percolation threshold is dramatically reduced with the additional advantage of preserving the bulk mechanical properties.

One such promising electrically conductive class of nano filler materials are polyaniline (D-PANI) nanofibers which are straightforward to synthesize [1], show decent electrical conductivity [2] and can be solution processed in common organic solvents. Moreover, the density and diameter control of D-PANI nanofibers grown in the presence of an insulating host material offers the promise of forming more interconnected nanofiber networks that would lower the percolation threshold of a composite.

In order to solution process these organic conductors, Cao, et al. discovered that chemically doping and complexing D-PANI with sulfonic acids, a resulting D-PANI-ES (polyaniline emeraldine salt) can be co-dissolved in m-cresol in various ratios with an insulating host polymer (e.g. polyimide) which forms robust, transparent conducting films when cast from

solution. Since then, other approaches have been introduced such as blending of D-PANI-ES through electrochemical [3,4] and chemical [5] polymerization of aniline monomer on polymer substrates; coating of D-PANI-ES polymer surfaces [6,7], dispersing of polyaniline in the melt of a thermoplastic polymer [8,9,10,11], blending undoped D-PANI with poly (vinyl pyrrolidone) then co-dissolving both polymers in N-methyl pyrrolidone (NMP) and subsequently doping in an acidic medium[12], dispersing of D-PANI-ES in a melt of thermoplastic polymer and blending the nonconducting form of PANI with polystyrene sulfonic acid [13]. The electrical conductivities of the blends obtained from these approaches varies, yet the conductive additive concentration is typically on the order of 10-16 vol. % to provide high conductivity above $10^{-8} \text{ S cm}^{-1}$. The processing of doped polyaniline and host polymer in a single solvent as developed by Cao et al. [14] gave high electrical conductivities at relatively low loading levels of polyaniline salt. The “nonconventional” relationship between electrical conductivity and polyaniline salt content in the solution cast blend was interpreted by many authors [15, 16, 17, 18] as evidence of a phase-separated structure with conducting networks or pathways of pure polyaniline salt forming at low loading levels. Potential conditions and interactions that may control the formation of the salt network and are thought to be operative in forming conductive pathways, are solvent-polymer interactions, polymer-polymer interactions, rate of crystallization of respective blend components, and kinetic effects of solvent evaporation from the blend solution.

In this work, the morphology of polyaniline nanofiber / polyimide nanocomposites were cast from NMP and subsequently characterized using small angle neutron scattering (SANS) techniques. Specifically, the blend morphology of D-PANI-0.5-CSA nanofibers in polyimide cast from NMP were studied and the results were compared to previously reported studies on a similar blend in polyimide [19]. In addition, it has previously been observed that D-PANI-0.5-CSA yields the highest electrical conductivity of all the surveyed polyaniline nanofibers [20].

2. Experimental

2.1 Synthesis and nanocomposite fabrication / characterization

Purification of F6 monomer: 4,4'-(hexafluoroisopropylidene) diphthalic anhydride (F6) (Lancaster, 99%) was purified by re-crystallization from acetic anhydride ($\geq 99\%$, Sigma-Aldrich). All procedures were carried out under nitrogen. Specifically, F6 (20 grams) was weighed into a dry 250 ml Erlenmeyer flask, followed by addition of 100 ml acetic anhydride ($\geq 99\%$, Sigma-Aldrich), and was stirred in a 100°C water bath until a clear solution was achieved. This solution was stored under N_2 , in the dark overnight. The resultant white crystals were filtered, washed with anhydrous ether (Sigma-Aldrich, $\geq 99.7\%$), and dried under high vacuum at 40°C overnight. Recrystallized F6 was determined to be pure by NMR (^1H : CDCl_3 , δ 8.143, δ 8.123, δ 8.033, δ 7.916, δ 7.896 ppm, ^{19}F δ 63.583 ppm).

Purification of ODA: All procedures were carried out under nitrogen. Anhydrous N,N-dimethyl formamide (Sigma-Aldrich 99.8 % , 22.5ml) was added to 4,4'-oxydianiline (ODA) (6.0 grams) and stirred until dissolved. Anhydrous toluene (Sigma-Aldrich 99.8 % , 225 ml) was heated in an oil bath at about 90°C and added to the above stirring solution. The solution was stirred in the oil bath at 90°C until clear, stored in the dark. The resultant white crystals were filtered, washed with toluene, and dried in a vacuum oven at 40°C overnight. Recrystallized ODA was determined pure by NMR (^1H : DMSO, δ 6.676, δ 6.647, δ 6.557, δ 6.528, δ 4.806 ppm).

ODA / F6 Copolymerization: In a N_2 -filled glovebox, a 50-mL RB-flask equipped with a stir bar was charged with 4,4'-oxydianiline (ODA) (0.104 grams, 5.20 mmol) followed by N,N'-dimethylacetamide (DMAc, Sigma-Aldrich $\geq 99\%$, distilled from BaO under high vacuum) solvent (5.0 ml) added with a gas tight syringe (Hamilton Co.) and allowed to stir until a clear

solution was obtained. A second 50-mL RB-flask equipped with a stir bar was charged with 4,4'-(hexafluoroisopropylidene) diphthalic anhydride (F6) (2.310 grams, 5.20 mmol) followed by addition of DMAc (4.0 ml). This mixture was allowed to stir for about 1 hour until the F6 monomer dissolved to a fine slurry. Using a gas-tight syringe, the F6 solution was added dropwise to the stirring ODA solution over 30 minutes. The F6-containing round bottom flask was rinsed with DMAc, which was added to the reaction vessel, for a total DMAc volume of 20 ml. The moderately viscous, transparent, and slightly yellow, poly(amic acid) solution was stirred overnight at room temperature, then stored under nitrogen in the freezer prior to PANI addition.

Synthesis of deuterated Polyaniline Nanofibers optimally doped with camphorsulfonic acid

(D-PANI-0.5 CSA): Aniline monomer (Aldrich Chemical) was vacuum distilled over zinc metal before use. 1R-(-)-10-camphorsulfonic acid (CSA) from Aldrich Chemical was used as received. The nanofiber form of PANI, designated as nanofiber PANI, was successfully polymerized (using a synthesis described in detail elsewhere [21]) from enriched monomer, allowing parts of the PANI chains to be “tagged” using deuterated hydrogen (D7, 98%) (Cambridge Isotopes). This molecular “tagging” was used in selected areas of the polymer backbone structure to improve the sensitivity of the SANS instrument and the polymer is designated as “D-PANI.”

Interfacial Polymerization: Interfacial polymerization is the method by which electrically conducting D-PANI nanofibers are created in the presence of a polymer system. Therefore, a biphasic method, or “interfacial polymerization,” was used to grow D-PANI nanofibers in the presence of the PI precursor, poly(amic acid) (PAA), which is the fundamental requirement for creating these nanofibers. This interfacial method requires a solvent in which the PI precursor, PAA, forms a biphasic layer in the presence of the polyaniline monomer, aniline. For this work,

PAAAs were dissolved in a polar aprotic solvent (dimethyl acetamide) and layered with the aniline monomer dissolved in carbon tetrachloride.

Using the above described technique, poly(amic acid) based on a fluorinated dianhydride monomer, 4,4'-(hexafluoroisopropylidene) diphthalic anhydride was chosen with the objective of obtaining maximum molecular interaction with D-PANI with minimum color in the fully cured PI. Using these fluorinated PAAAs, a series of D-PANI/PAA nanocomposites were synthesized and characterized before PAA was thermally converted to the fully cured PI. The optimal range of D-PANI concentration in PAA necessary to achieve both high transparency in the visible region and high conductivity in the final PI, was found to be between 0.2 and 0.5% (wt/wt) before the onset of absorption at 500 nm was seen. These D-PANI-based nanocomposite films of 3-4 micron thickness were pinhole-free and very homogeneous. Infrared (IR) spectra of the light amber-colored composite were consistent with D-PANI embedded PI. Electron microscopy of the fractured side surface of the D-PANI polyimide film revealed D-PANI fibers with diameters <100 nm protruding from inside the bulk polyimide and which appeared to be enmeshed within the PI matrix. Increasing the D-PANI concentration beyond the 0.5% (wt/wt) resulted in a corresponding absorbance in the visible region at 500 nm.

When D-PANI is mechanically mixed at these same loading values, higher conductivity values are seen, but with increased absorbance in the 500-nm peak. IR spectroscopy was used to determine the degree of conversion from the poly (amic acids) to the subsequent polyimides. At a 0.5% D-PANI nanofiber loading, a 91% conversion of PAA to PI occurred after a cure of 80° C for 4 hours, 120° C for 1 hour, 200° C 1 hour, and 275° C for 1 hour without sacrificing optical transmission in the visible range (2% absorbance at the 500-nm benchmark peak) or electrical conductivity (1.6×10^{-6} S/cm). In general, higher cure temperatures above the optimal 275° C value yielded small gains in percent conversion to PI from PAA, but dramatically

decreased both conductivity and transparency of D-PANI/PI nanocomposite. Since both conductivity and optical clarity are related to percent conversion of PAA to PI and nanofiber loading, these nanocomposites require a compromise between these two variables.

2.2. SANS Characterization

The small angle neutron scattering experiments (SANS) were performed using the BT5 thermal neutron double-crystal instrument at the National Institute of Standards and Technology (NIST), NCNR (NIST) Center for Neutron Research in Gaithersburg, MD, USA. The samples were run for 6-8 hours each with path length varying from 0.25 -1mm sample thickness in quartz cells at a neutron wavelength of 2.4 Å. The background from an empty cell run was subtracted from all the data, and the subtracted data processed to an absolute scale by use of the direct beam intensities. A typical run allowed measurements with $0.00003 < Q_h \text{ Å}^{-1} < 0.005$, where Q_h is the wavevector component in the horizontal plane. This corresponds to probing length scales from 1500 -200,000 Å. The instrument has a slit beam profile extended in the vertical direction corresponding to a Q_y width of 0.117 Å^{-1} . The observed beam intensity at any nominal Q_h is thus a convolution of a range of actual Q values [22]. The observed data can be de-smeared in order to simulate what would be seen by a pinhole beam method [23] or a scattering model smeared with the resolution function of the instrument as part of the fitting procedure. The latter is preferred as it is more reliably applicable and the data presented here have not been de-smeared.

3. Results and Discussion

The neutron scattering results for the polyimide blends could not be fitted to the Ornstein-Zernike/Zimm functional form [24, 25], indicating that the D-PANI and PI polymer chains do not mix at the molecular level. Phase separation is corroborated by SEM images seen in Figure 1.

The presence of a lamellar-type polyimide peak ($q = 0.022 \text{ \AA}^{-1}$) and a peak corresponding to a spacing of about 1.5 nm ($q \approx 0.01 \text{ \AA}^{-1}$) in the composite can be seen in the small angle X-ray scattering (SAXS) profiles in Figure 2. The maximum of this former peak is associated with the distance between centers of adjacent polyimide chains and both shifts in position and a decrease in intensity are seen. The decrease of this peak ($q = 0.022 \text{ \AA}^{-1}$) when 1% PANI salt is added suggests that PANI suppresses the development of any order in the PI host, which is beyond simple volume dilution. This suppression behavior is consistent with other PANI blends systems.[22] Interpretation of this shift to lower q values means that the conformationally dissimilar salt (PANI chains) penetrates the semicrystalline regions of PI.

As seen in Figure 3, the data for both 1 and 2% (wt/wt) D-PANI-0.5-CSA in polyimide blend can be fitted by the D-B model treatment of the SANS blend data. The good fit to this model indicates that the D-PANI-0.5-CSA phase has neither a volume fractal nor a surface fractal nature. A correlation length of 21 nm was determined and the transverse lengths are 22 and 70 nm for the D-PANI-0.5-CSA and polyimide phases, respectively. From this, we were able to calculate the correlation length of 71 nm and the transverse lengths of 115 nm for D-PANI-0.5-CSA and 187 nm for neat polyimide. These data are also well represented by the inverse power law with an exponent of approximately 4 (3.99) which corresponds to the limit of the D-B model for large correlation lengths. Increasing the PANI salt concentration to 5 vol. % (Figure 3) results in some small deviations from the D-B model at the lower momentum transfer (q) values. Furthermore, relative to the smaller D-PANI-0.5-CSA loading volumes of 1, 2% (wt/wt), the size of both the D-PANI-0.5-CSA and the polyimide domains increased, which indicates that the D-PANI-0.5-CSA tended to aggregate and become less dispersed.

4. Conclusions

The immiscible nature of the conformationally dissimilar polyaniline nanofiber and polyimide host is established by a series of experiments involving neutron and X-ray scattering. Based on these techniques, we conclude that the crystal structure of the polyimides is not disrupted, and that there is no mixing on a molecular level between the two components. The morphology of the conducting salt component was analyzed by SANS data and was treated by two common models: Debye-Bueche (D-B) and inverse power law (IPL). Due to deviations in the linear curve fitting over a large scattering range, neither the D-B nor the IPL model could be used to characterize the size and shape of all D-PANI-0.5-CSA/polyimide blend systems. At 1 and 2% concentration, the D-B model suggested D-PANI-CSA salt domains between 20 and 70 Å with fractal geometries implied by the IPL model. As salt concentrations are increased to 5%, the structures are observed to change, but there is no simple structural model that provides a suitable basis for comparison.

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Fig. 1. Scanning electron microscope (SEM) images of 1% (wt/wt) D-PANI-0.5-CSA nanofiber/polyimide composite post solvent evaporation fracture and synthesized by interfacial synthesis route: (a) cracked film, (b) view inside a crack, exposing the bulk of the composite film to show (c) and (d) D-PANI nanofibers (light color) seen in wall of nanocomposite (50 nm in diameter) surrounded by the insulating, low emissive (dark color) host polyimide.

Fig. 2. Small Angle X-ray Scattering (SAXS) plot for (a) unfilled, neat polyimide (0% D-PANI) and (b) 1% (wt/wt) D-PANI-0.5-CSA/polyimide blend. The presence of a lamellar-type polyimide peak is seen in the unfilled polyimide, but disappears upon a 1% salt (i.e. D-PANI-0.5-CSA) loading.

Fig. 3. SANS blend data of 0% (neat) 1%, 2% and 5% D-PANI-0.5-CSA nanofibers/polyimide blends.

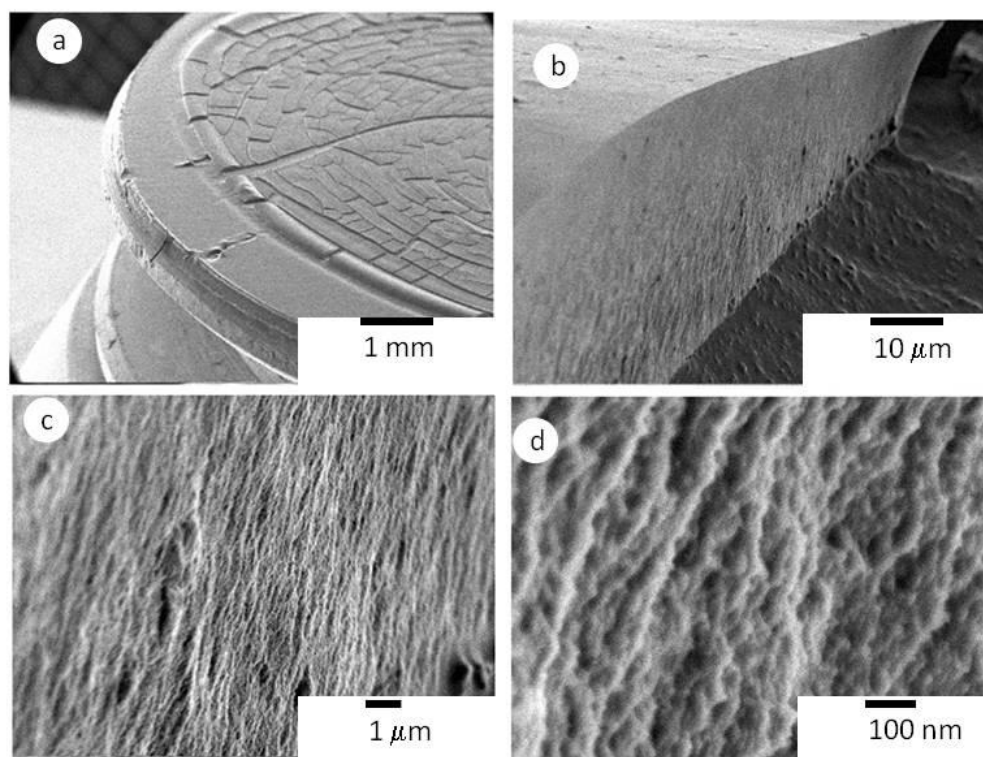
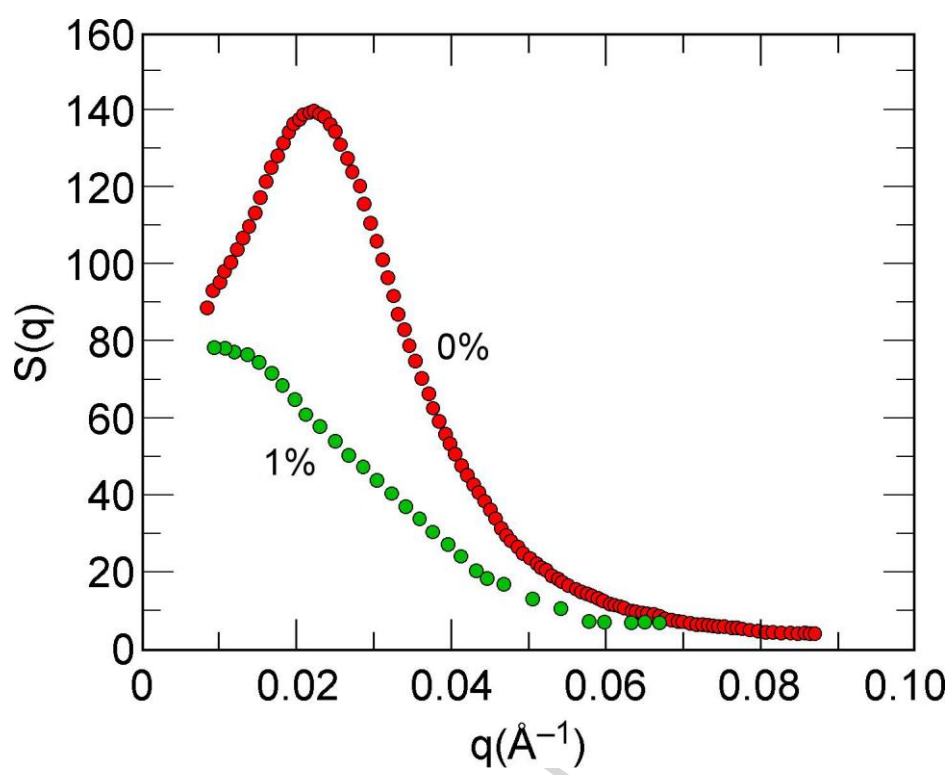
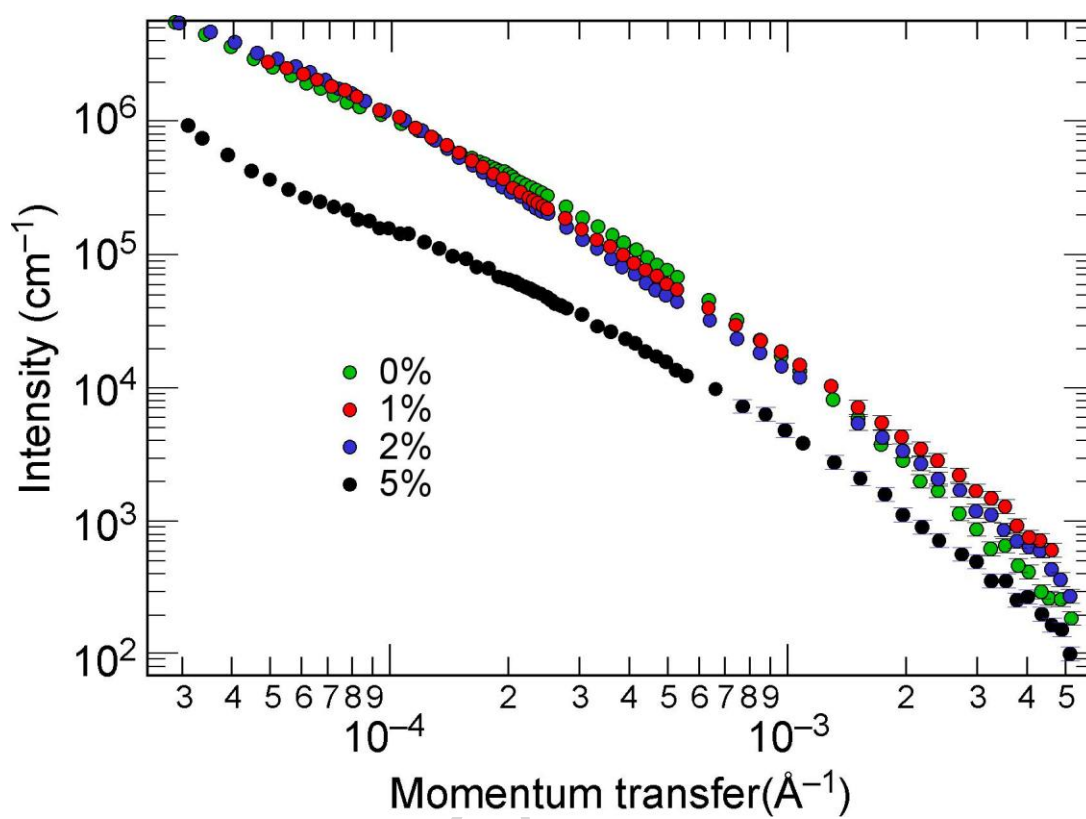


Fig. 1

**Fig. 2**

**Fig. 3**

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